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Indian Standard

CHROMITE SAND FOR FOUNDRIES — METHODS OF CHEMICAL ANALYSIS

PART 2 DETERMINATION OF IRON

भारतीय मानक

फाउँड्रियों के लिए क्रोमाइट सैंड — रासायनिक विश्लेषण

भाग 2 लोह ज्ञात करना

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

October 1989 **Price Group 1**

Methods of chemical Analysis of Ores, Minerals and Refractories Sectional Committee, SMDC 35

FOREWORD

This Indian Standard (Part 2) was adopted by the Bureau of Indian Standards on 29 March 1989, after the draft finalized by the methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee had been approved by the Structural and Metals Division Council.

IS 6788 : 1973 'Specification for chromite sand for foundries' covers the requirements for the various constituents. As chromite sand is widely used in steel/iron castings, a need was felt to prepare the standard on methods of test based on requirements of IS 6788 : 1973.

This standard has been prepared in different parts. This part (Part 2) covers the chemical analysis of iron, and other parts are:

Part 1 Determination of silica

Part 3 Determination of chromium

Part 4 Determination of calcium

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHROMITE SAND FOR FOUNDRIES — METHODS OF CHEMICAL ANALYSIS

PART 2 DETERMINATION OF IRON

1 SCOPE

1.1 This standard (Part 2) covers a method for determination of total iron (as Fe_2O_3) in chromite sand for foundries.

2 REFERENCE

IS 1811 : 1984 Methods of sampling foundry sands (*first revision*)

3 SAMPLING

3.1 The sample shall be drawn by the method prescribed in IS 1811 : 1985.

3.2 Pulverize about 10 g of sample in an agate mortar to pass through 75 µm IS test sieve. Dry at 105 to 110°C for 2 hours.

4 GENERAL

4.1 Use of Filter Papers

In this method, only relative number of Whatman filter paper has been prescribed since they are commonly used. However, any other suitable brand of filter paper of corresponding porosity and equivalent quality may also be used.

5 QUALITY OF REAGENTS

5.1 Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

6 DETERMINATION OF IRON BY DICHROMATE TITRATION METHOD

6.1 Outline of the Method

Sample is fused with sodium peroxide and the mass extracted with water is filtered. The residue is dissolved in hydrochloric acid and iron is reduced. The reduced iron is titrated with standard potassium dichromate solution in acidic medium.

6.2 Reagents

6.2.1 *Sodium Peroxide*, granular.

6.2.2 *Dilute Hydrochloric Acid*, 1 : 1 (*v/v*).

6.2.3 *Ammonium Chloride Solution*, solid.

6.2.4 *Ammonium Hydroxide*, rd = 0·90.

6.2.5 *Stannous Chloride Solution*

Dissolve 10 g of pure stannous chloride in 10 ml concentrated hydrochloric acid and boil till clear solution is obtained. Cool and dilute to 100 ml. Keep in an amber coloured bottle.

6.2.6 *Mercuric Chloride Solution*

Prepare a saturated solution of mercuric chloride in water.

6.2.7 *Sulphuric-Phosphoric Acid Mixture*

Add slowly 150 ml of concentrated sulphuric acid (rd = 1·84) to 700 ml water with continuous stirring. Add to this, 150 ml syrupy phosphoric acid (85 percent). Destroy any oxidizable impurity by adding potassium permanganate solution (0·1 N) dropwise till the pink colour persists. Cool the solution.

6.2.8 *Sodium Diphenylamine Sulphonate Indicator Solution*

Dissolve 0·32 g of barium diphenylamine sulphonate in 100 ml hot water. Add 0·5 g of sodium sulphate, stir, and filter off the precipitate of barium sulphate. Store in a dark coloured bottle.

6.2.9 *Standard Potassium Dichromate Solution* (0·1 N)

Dry pure crystals of potassium dichromate (AR/GR quality) in an air oven at 105 to 110°C for an hour. Cool in a desiccator and transfer carefully 4·903 g through a funnel to a one litre volumetric flask using water at room temperature. Wash the funnel and dissolve the salt by shaking. Make up to mark.

6.3 Procedure

6.3.1 Fuse 0·500 g of sample with 4 g sodium peroxide in a nickel crucible of 30 ml capacity provided with lid. Keep the fused mass at dull red heat with occasional swirling for around ten minutes to ensure complete reaction. Cool to room temperature and keep in a 400 ml beaker containing around 50 ml of water. The beaker is covered with a watch glass and the contents of the crucible allowed to react with water. Wash out the crucible and lid with water in to the beaker. Dilute to around 100 ml and heat to boil. Keep boiling the extract for ten minutes to destroy the hydrogen peroxide. Cool and filter through Whatman filter paper No. 540 with pulp. Wash the residue with 1 percent hot sodium carbonate solution. Preserve the filtrate (*A*) for chromium determination.

6.3.2 Dissolve the residue in hot dilute hydrochloric acid and reprecipitate iron hydroxide by addition of 5 to 7 g of ammonium chloride followed by ammonium hydroxide till distinctly ammonical. Boil for 5 minutes and digest on a steam bath for 20 minutes and filter through Whatman filter paper 40 and wash the residue well with hot wash solution (containing 1 percent ammonium chloride made distinctly ammonical) till free from chloride. Add the filtrate to filtrate (*A*) and set aside the combined filtrate (*B*) for chromium determination.

6.3.3 Dissolve the reprecipitated ferric hydroxide in hot dilute hydrochloric acid, cool and make up to 250 ml in a volumetric flask with water and shake well to homogenize. Take an aliquot from this solution, heat to boiling and to the boiling solution, add stannous chloride solution dropwise with continuous stirring until the solution just becomes colourless, add 1 drop in excess. Cool rapidly, add 5 ml of mercuric chloride solution and shake to destroy excess stannous chloride. At this stage, a silky white precipitate appears.

6.3.4 Add 15 ml of sulphuric-phosphoric acid mixture, and dilute to about 200 ml. Add three drops of sodium diphenylamine sulphonate indicator and titrate slowly with standard potassium dichromate solution, stirring well. Continue titration carefully till a drop of dichromate solution gives a permanent violet-blue colouration.

6.4 Calculation

$$\text{Total iron (as } Fe_2O_3 \text{), percent} = \frac{A \times B \times 0{\cdot}079\,86}{C} \times 100$$

where

A = volume in ml of standard dichromate solution consumed,

B = normality of standard patassium dichromate solution, and

C = mass in g of the sample represented by the aliquot taken.

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